

SUPERCONDUCTING ELECTRONIC FILM STRUCTURES(U)  
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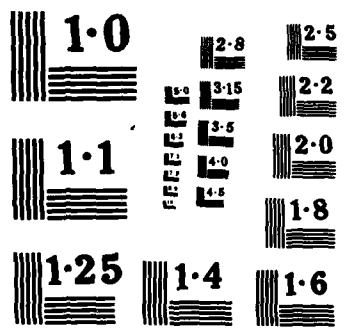
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FINAL REPORT

AFOSR-TR- 85 - 0461

January 1, 1983 to December 31, 1984

SUPERCONDUCTING ELECTRONIC  
FILM STRUCTURES

By

A. I. Braginski and J. R. Gavalier

Westinghouse Electric Corporation  
Research and Development Center  
Pittsburgh, Pennsylvania 15235

AFOSR Contract No. F49620-83-C-0035

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fluctuations in thickness across each junction area which appear to be a universal property of tunnel barriers. Single crystal films evaporated from ultrahigh purity Nb ( $< 10$  ppm interstitials) were prepared for rf loss studies.

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1. Final Report, Superconducting Electronic Film Structures

January 1, 1983 to December 31, 1984

AFOSR Contract No. F49620-83-C-0035

A. I. Braginski and J. R. Gvaler.



## 2. ABSTRACT

Progress toward the fulfillment of the five main objectives of this program include the following results: V-Si and Nb-Ge films with critical temperatures of  $\sim 12\text{K}$  were reactively sputtered at deposition temperatures of  $< 500^\circ\text{C}$ . In the V-Si films the A15 phase was retained at temperatures of  $< 300^\circ\text{C}$ . NbN film with critical temperatures of  $12\text{K}$  were magnetron reactively sputtered on  $20^\circ\text{C}$  substrates. Critical temperatures of  $> 15\text{K}$  were obtained on  $300^\circ\text{C}$  substrates. Solid state epitaxial growth of NbN single crystals was achieved on six different surface orientations of sapphire by annealing sputtered amorphous Nb-N. Epitaxial films of Nb, Nb-Sn, and Nb-Ge were e-beam evaporated on sapphire. A LEED study of Nb-Ir single crystals lead to an in situ procedure for producing epitaxial quality surfaces. A new UHV facility capable of e-beam evaporation and magnetron sputtering of films and equipped with in situ RHEED, XPS, and Auger spectroscopy was installed and became operational. An XPS study of tunnel barrier thickness discovered large fluctuations in thickness across each junction area which appear to be a universal property of tunnel barriers. Single crystal films evaporated from ultrahigh purity Nb ( $< 10\text{ ppm}$  interstitials) were prepared for rf loss studies.

### 3. OBJECTIVES

The objectives of the Westinghouse-AFOSR program are:

1. Investigate the low-temperature synthesis of high-critical-temperature superconducting films.
2. Grow epitaxially single-crystal superconducting films and coherent layered structures.
3. Characterize the near-surface crystalline perfection of superconducting layers and their interfaces by in situ methods.
4. Study tunnelling into high-critical-temperature superconducting films and other electronic film properties.
5. Explore electric characteristics of layered film structures.

## 4. ACCOMPLISHMENTS

### 4.1 Preamble

This five-year research program was initiated in January 1983. It is aimed at understanding and improving the superconducting and normal state properties of layered, epitaxial, thin film structures incorporating high-critical-temperature superconductors. Anticipated results are intended to form a material science base for a future technology of high-operating temperature superconductors. This report serves as the Final Report covering the period from January 1, 1983 to December 31, 1984. Under a new contract, beginning on January 1, 1985, the program will continue. The present status of studies initiated in 1983-1984 is described in this report.

Successful performance under the program has required the purchase of a new type of ultra-high-vacuum (UHV) deposition and in situ analytical facility, hereafter referred to as the Superlattice Analytical and Deposition Facility (SDAF). This facility was delivered by Riber S.A. of France in the latter part of 1983. Its installation was largely completed by mid-1984 and experimental work is proceeding. Highlights of this work are presented below.

### 4.2 Low-Temperature Synthesis of High- $T_c$ Films

Low-temperature synthesis of high- $T_c$  superconducting films is required for S-I-S tunnel junction fabrication to avoid barrier damage. It is also of considerable scientific interest to further the understanding of stable and metastable compound formation. Progress has been made in this area both with Al<sub>5</sub> and B1 structure material. These will be considered separately.

#### 4.2.1 Al5 Structure Compounds

With the SDAF operational, work on impurity-influenced low-temperature diffusion reactions between A and B elements to form  $A_3B$  Al5-structure compounds is now in progress. The first phase of this investigation has involved the formation of Nb-Sn diffusion couples (bilayers) containing controlled amounts of oxygen which are then annealed to form  $Nb_3Sn$ . Ultra-pure niobium single crystals (less than 10 ppm of interstitials) were especially prepared for this study by the Max Planck Institute in Stuttgart, West Germany. Using this starting material, niobium films were deposited at 850°C on epitaxially polished sapphire substrates. These films showed atomically smooth surfaces and single crystal diffraction patterns when viewed with in situ RHEED, immediately after deposition. The Sn layers deposited on top were also single-crystalline. Polycrystalline Nb and Sn bilayers were obtained by depositing niobium onto non-epitaxially polished sapphire at room temperature. Controlled amounts of oxygen were introduced into some of each of these two types of films. The level of oxidation was monitored by in situ XPS. Annealing of the pure and the oxygen-doped Nb-Sn diffusion couples was done at pressures of  $< 4 \times 10^{-11}$  torr and at temperatures of 650°, 700°, and 850°C for times of 30 minutes or five hours. The samples have been characterized by four-point resistance measurements to determine the critical temperature,  $T_c$ , and resistivity vs temperature, and by Auger spectroscopy to determine depth profiles of interdiffusing elements. Results to date suggest that:

1. The presence of grain boundaries lowers the temperature required for Al5 phase formation.
2. Oxygen seems to enhance the growth of a more stoichiometric Al5 phase when grain boundaries are present.
3. In the case of single crystal Nb films the addition of oxygen has no positive effect on Al5 growth.

Satisfactory explanations of these observations are not yet in hand. A summary of the initial results will be presented at the March 1985 Meeting

of the American Physical Society (APS). This work is, however, still in its initial stages, and will have to continue into the following years.

An advance in the low-temperature synthesis of Al<sub>5</sub> compounds was achieved by reactively sputtering V<sub>3</sub>Si and Nb<sub>3</sub>Ge films. In 1983 this was done in a standard dc diode system. In 1984 these compounds have again been prepared by reactive sputtering but this time using a magnetron system. Although the new SDAF includes a UHV magnetron sputtering capability, a second much simpler magnetron system was used for these experiments. The reactive sputtering of V<sub>3</sub>Si and Nb<sub>3</sub>Ge requires the use of SiH<sub>4</sub> and GeH<sub>4</sub> gas and, in the case of Nb<sub>3</sub>Ge, a background impurity level of the order of 10<sup>-6</sup> torr. Since the introduction of this type of contamination into the SDAF was deemed unadvisable, this second magnetron system was constructed (at no cost to AFOSR). This system has a single U. S. Gun I sputtering head, a high-temperature substrate heater, and a mixing chamber for preparing the desired GeH<sub>4</sub>/Argon and SiH<sub>4</sub>/Argon mixtures. The system uses diffusion pumping and has a background impurity level in the low 10<sup>-6</sup> torr region. Using magnetron sputtering V-Si and Nb-Ge films were prepared which were found to be dramatically different from those deposited in the dc diode system. V<sub>3</sub>Si films prepared in the magnetron system crystallized into the Al<sub>5</sub> structure at deposition temperature as low as 290°C compared to the previous minimum temperature of ~ 450°C. These films were superconducting with T<sub>c</sub>'s of ~ 7K. A second significant observation was that the dependences of critical temperature (measured resistively) on film composition were very different. In the case of the V-Si films that were dc diode sputtered, near optimum T<sub>c</sub>'s of ~ 16K were found in films having average compositions ranging from V<sub>83</sub>Si<sub>17</sub> to V<sub>64</sub>Si<sub>36</sub>. Studies on bulk samples have shown that such high critical temperatures are obtained in the V-Si system only at compositions at or very near to V<sub>75</sub>Si<sub>25</sub>. In the case of the magnetron sputtered V-Si, T<sub>c</sub> peaked in films having 3/1 stoichiometry and dropped sharply on either side of this composition, similar to bulk data. The chemical compositions of these films have been determined on a microscale by energy dispersive X-ray spectroscopy in a scanning transmission electron microscope using a 50 Å beam. Results show that the high-T<sub>c</sub>'s found in films

whose average compositions are far removed from the ideal  $A_3B$  are due to these films having regions of 3/1 stoichiometric material incorporated in a matrix of non-3/1 material. As an example, approximately 70% of the grains in a high- $T_c$  V-Si film were found to have a composition of  $\sim V_{75}Si_{25}$  while the remainder were  $\sim V_{95}Si_{05}$ . The grains having 3/1 stoichiometry were interconnected and thus the resistively measured  $T_c$  was high (16.8K), much higher than would be expected based on the average composition of the film. These analytical results also show that the Al<sub>5</sub>V<sub>75</sub>Si<sub>25</sub> films grown at < 300°C deposition temperatures are homogeneous on a microscale. The low  $T_c$ 's ( $\sim 7K$ ) of these films must therefore be attributed to disorder rather than improper stoichiometry. Most of these data were presented at the Applied Superconductivity Conference, San Diego, CA (September 1984). The more recent data on the microchemical analyses of these films will be reported at the March 1985 APS Meeting.

#### 4.2.2 B1 Structure Compounds

As discussed above, Al<sub>5</sub> structure superconductors can be grown at temperatures below 300°C. These films, however, were found to have low  $T_c$ 's, despite having proper 3/1 stoichiometry. The low  $T_c$ 's were attributed to structural disorder. In the B1 structure superconductors it has been well established that disorder has only a minimal effect on  $T_c$ . For this reason a large amount of research on the low-temperature growth of B1 structure superconductors has been done by various investigators. In past Westinghouse-AFOSR programs extensive work has been done on the preparation and properties of the B1 superconductor, NbN. Drawing on this experience, conditions have been established for magnetron sputtering of polycrystalline NbN at low temperatures ( $\leq 300^\circ C$ ) using the SDAF. These films which have  $T_c$ 's of up to 15K are now being used for tunnelling experiments. In the low-temperature growth of polycrystalline NbN on MgO epitaxial effects have been observed. This work will be presented at the 1985 CEC-ICMC Conference.

#### 4.3 Epitaxial Growth of Superconducting Films

The investigation of epitaxial growth processes has a technological as well as a scientific motivation. Elimination of near-interface structural disorder in layered film structures will make high- $T_c$  S-I-S tunnel junctions possible. The so called "polycrystalline epitaxy" has been shown to be useful for stabilizing  $Nb_3Ge$  in its high- $T_c$  form. Finally, the availability of single crystals of high- $T_c$  superconductors will permit the investigation of their intrinsic properties and will advance the science of superconductivity.

During 1983 solid-state epitaxial growth of NbN on sapphire substrates was achieved, representing a major achievement of this program. This study was continued during 1984. Single crystal NbN films have now been grown on sapphire with  $(1\bar{1}02)$ ,  $(0001)$ ,  $(10\bar{1}0)$ ,  $(11\bar{2}0)$ , and  $(\bar{1}012)$  surface orientations as well as the initially reported  $(2\bar{1}\bar{1}3)$ . NbN films were sputtered on these substrates after various surface treatments. The results of these experiments clearly indicate that surface preparation and cleanliness are the critical factors for obtaining single crystal epitaxial growth on sapphire.

An effort was initiated to determine whether high- $T_c$  NbN can be grown epitaxially on MgO, a potential junction barrier material. The MgO crystallizes in the B1 structure and has a lattice parameter close to that of NbN. Efforts will be continuing to study both polycrystalline and single crystal epitaxy of NbN on MgO. A presentation of initial results on the NbN single crystal growth on sapphire was made at the March 1984 APS Meeting. A paper, which also includes more recent data, was presented at the 1984 LT-17 Conference and has been published in the proceedings.

Work on epitaxial Al5 films is centering on  $Nb_3Ge$  and  $Nb_3Sn$ . Single crystals of  $Nb_3Ir$  for epi-substrates were obtained at no major cost to the program. The  $Nb_3Ir$  should be an ideal epitaxial substrate material because of its Al5 structure and lattice parameter similar to that of  $Nb_3Ge$ . Two single-crystal  $Nb_3Ir$  rods, 6 mm in diameter and with (100)

major axis, were prepared in 1983 for use in this program through the courtesy of Dr. Eric Walker, using the crystal growing and zone refining facilities at the University of Geneva in Switzerland. Raw materials for the synthesis were supplied by this program.

During 1983 the characterization of  $\text{Nb}_3\text{Ir}$  rods was done using X-rays and the electron microprobe. These appear to be the first high-quality  $\text{Nb}_3\text{Ir}$  crystals ever grown. In 1984 a method for epitaxial  $\text{Nb}_3\text{Ir}$  substrate preparation has been developed based on XPS characterization, reflection electron diffraction (RHEED), and especially low energy electron diffraction (LEED) coupled with Auger spectroscopy. The LEED/Auger technique permitted one to determine in-situ substrate processing conditions that for (100), (111), and (110) substrates result in a perfect  $1 \times 1$  diffraction pattern representing the top few atomic monolayers. Surface reconstruction effects have also been observed by LEED and attributed to the injection of oxygen impurity resulting in Nb-suboxide formation. The (111) plane is less susceptible to reconstruction than (100). Results to date will be presented at the March 1985 APS Meeting.

An activity parallel to that described above has been conducted to study yttria-doped  $\text{ZrO}_2$  substrate crystals. These crystals have a lattice parameter compatible with  $\text{Nb}_3\text{Ge}$  but a different crystal structure (that of calcium fluoride). The LEED/Auger investigation has shown in this case that the  $\text{ZrO}_2$  surface decomposes in UHV upon heating. Consequently, no epitaxial quality substrates could be obtained to date. Preliminary work was initiated on calcium fluoride ( $\text{CaF}_2$ ) itself. The  $\text{CaF}_2$  and  $\text{Nb}_3\text{Sn}$  have an acceptable lattice parameter match, so that calcium fluoride (and, possibly, other fluorides) could be attractive as a buffer substrate and epitaxial barrier material. The LEED/Auger study has confirmed that  $\text{CaF}_2$  grows epitaxially on (11 $\bar{2}$ 0) sapphire. Consequently, sapphire/ $\text{CaF}_2$  substrates for  $\text{Nb}_3\text{Sn}$  growth are being fabricated. A study of  $\text{Nb}_3\text{Ge}$  and  $\text{Nb}_3\text{Sn}$  epitaxy on  $\text{Nb}_3\text{Ir}$  and  $\text{CaF}_2$  substrates is planned for 1985-1986.

An investigation of  $\text{Nb}_3\text{Ge}$  and  $\text{Nb}_3\text{Sn}$  epitaxial growth directly on sapphire is in progress. In the UHV environment, and a virtual absence



of oxygen impurity, the high- $T_c$   $Nb_3Ge$  is not expected to form. Indeed, on non-epitaxial sapphire only the Ge-deficient polycrystalline A15 phase with  $T_c \approx 6K$  was obtained. On epitaxial  $(11\bar{2}0)$  sapphire, however, films deposited in identical conditions exhibited epitaxial RHEED pattern and had a  $T_c \geq 13K$  thus suggesting the stabilization of a Ge-richer A15. The  $Nb_3Sn$  films on  $(11\bar{2}0)$  sapphire have shown a very high degree of orientation and the effect of "phase locking" thus confirming the current results obtained at Stanford University under another AFOSR contract. An X-ray rocking-curve study to determine the single crystal quality is in progress. To complement the study of epitaxial B1 and A15 layers, single crystal films of niobium have also been fabricated in SDAF using sapphire substrates of various orientation. The single-crystalline nature of these atomically-smooth films has been determined by in-situ RHEED and X-ray rocking curves. These films will be used in studies described in Section 4.2, in studies of RF surface losses (in collaboration with the MIT-Lincoln Laboratory), and in a spectroscopic [ $\alpha^2 F(\omega)$ ] study of tunneling anisotropy. First shipment of such samples to MIT occurred in July 1984.

#### 4.4 Characterization of Near-Surface Layers

The purpose of this task is to develop and apply methods of surface and interface characterization that are appropriate for the in-situ investigation of thin films and layered structures generated under other tasks of the program. Crystallinity, phase composition, and physical uniformity are of particular interest.

Performance of this task required an extensive use of the in-situ analytical equipment of the SDAF. RHEED measurements were routinely used to examine the quality of surface preparation of single-crystal substrates prepared in vacuum, the crystallinity and orientation of deposits after an initial layer had been formed ( $< 100 \text{ \AA}$ ) and after the deposition was completed. The ex-situ LEED permitted a refinement in determining the  $Nb_3Ir$ ,  $ZrO_2$  and  $MgO$  epitaxial substrate quality up to the first couple of monolayers, and the development of effective surface

preparation methods. In-situ X-ray photoelectron spectroscopy (XPS) was used to measure the thickness of oxidized metal overlayers before counterelectrode deposition, to determine the composition of alloy and compound films before subsequent layers were deposited, and to monitor the removal of contaminants and surface oxides during substrate surface preparation.

The surface analysis capability of the SDAF was not fully operational until the last quarter of 1984 when the data acquisition software was delivered to Westinghouse. A separate surface analysis facility (VC Escalab Mark II) was used for a number of measurements which did not require the advantages of in-situ analysis.

The interface between a superconducting electrode and an oxidized metal tunnelling barrier was studied with Nb, Nb<sub>3</sub>Sn, and NbN base electrodes, and oxidized aluminum, yttrium, and magnesium barriers. The crystallinity of the barrier was determined to compare with the results of tunnelling measurements. For junctions with Nb base electrodes, the barrier crystallinity could be prescribed to be highly textured by using a single-crystal base electrode formed at a high substrate temperature, randomly-oriented polycrystalline by depositing on polycrystalline Nb, or amorphous by ion-milling the barrier with low energy argon ions after oxidation.

For the first time, the thickness of tunnel barriers was measured by XPS as a function of the angle of photoelectron detection. A barrier layer of uniform thickness on a smooth base would be expected to have a measured thickness which was independent of angle. The apparent thickness-versus-angle dependence for a layer with a Gaussian distribution of thickness characterized by a particular value of the standard deviation, can be calculated to compare with experimental results.

Measurements were made on both Westinghouse and Stanford University ultrathin films and barriers. The smoothest ultrathin films in existence are those of amorphous Mo-Ge, 20Å thick, that have been studied at Stanford University. These have been used to separate the effects of

non-uniform thickness from the effects of a rough substrate. All tunnel barriers measured had a non-uniform thickness with a standard deviation comparable to the average thickness. The electrical properties of tunnel junctions must be dominated by the thinnest regions of the barrier, that is, the regions with the highest conductance. Consideration of these results leads to the conclusion that tunnelling characteristics are determined by a small fraction of the junction area for a broad variety of barrier materials formed by sputtering, by evaporation, or by thermal oxidation of a native oxide. Consequently, one of the directions of work aimed at improvements in junction reproducibility must be to study parameters improving barrier thickness uniformity.

A paper describing some of the work on superconductor-barrier interfaces was presented at the 1984 Applied Superconductivity Conference.

#### 4.5 Tunnelling

The study of tunnelling into high- $T_c$  superconductors represents a task of direct technological consequence while at the same time providing information about the intrinsic properties of the superconductor near its surface.

A comparison has been made between the leakage current of artificial tunnelling barriers formed by sputtering and by evaporation. Even for overlayers with similar crystallinity and which are equally effective at protecting the base electrode from oxidation, as judged by XPS, sputtered barriers on Nb had a lower leakage than evaporated barriers. However, evaporated barriers were very effective when used with  $Nb_3Sn$  base electrodes. An additional method of characterizing the properties of the barrier such as the flicker noise spectroscopy coupled with in-situ LEED must be used to understand the differences in leakage current. This is planned for 1985-1986.

Tunnelling has been used as a diagnostic of superconductor homogeneity. It is a sensitive probe of any electronic states below the superconducting gap energy which effectively shunt the junction and are

manifested as subgap conductance. The rise in current at the gap voltage for NbN-based junctions has been as sharp as for any NbN junctions in the literature indicating that even at a substrate temperature of 300°C, the material is homogeneous. For Nb<sub>3</sub>Sn-based junctions, the rise has also been as sharp as any reported, but the gap width could not be reduced below 0.6 mV. If the gap width of Nb<sub>3</sub>Sn is due entirely to inhomogeneity, the transition temperature width would have to be 3K. Resistive transitions, which are a poor indicator of homogeneity, were typically 0.1K wide.

Instrumentation for the tunnelling spectroscopy was constructed in 1984 and is presently undergoing testing. Also during 1984 work has been performed on adapting a computer program (obtained from Stanford University and Naval Research Laboratory) for the determination of the  $\alpha^2 F(\omega)$  parameter from current-voltage characteristics of tunnel junctions with proximity layers.

#### 4.6 Exploration of Electrical Characteristics of Layered Films

No work was performed toward this objective, as it was planned for the final period of the five-year program. For the continuation of the program in 1985 to 1987 this objective was narrowed to center on the study of RF surface losses in high-T<sub>c</sub> materials. The study will be performed in collaboration with the MIT-Lincoln Laboratory and Stanford University. This will provide a materials research backup for the development of high frequency analog signal processing devices.

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S. J. Bending, accepted for publication in IEEE Trans. on Magnetics.

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\* All or portions of these papers were prepared during the previous contract period.

## 6. PERSONNEL

M. Ashkin

A. I. Braginski

J. R. Gavalier

} Principal Co-Investigators

J. Gregg

M. A. Janocko

J. Schreurs

J. Talvacchio

## 7. COUPLING ACTIVITIES\*

1. "Potential of B1 Superconductors for High-Field Fusion Applications: Some New Results," A. I. Braginski, M. Ashkin, J. R. Gavalier, and J. Gregg, invited talk at the US-Japan Workshop on Superconducting Materials for Fusion, San Diego, May 23-27, 1983.
2. Participation in the NATO Advanced Study Institute on "Percolation, Localization and Superconductivity," A. I. Braginski, Les Arcs, France, June 19-July 1, 1983.
3. Participation in the CEC/ICMC (International Cryogenic Materials Conference), A. I. Braginski, Colorado Springs, August 15-19, 1983.
4. "Thin Film Materials," A. I. Braginski, invited presentation at the Workshop on Problems in Superconductivity, Copper Mountain, Colorado, August 21-23, 1983.
5. "Epitaxial Growth of B1 Structure NbN on Sapphire," J. R. Gavalier, J. Gregg, and J. Schreurs, contributed talk, March 1984 Meeting, American Physical Society.
6. "High- $T_c$  Superconducting Materials for Electronic Applications: Critical Issues," A. I. Braginski and J. Talvacchio, invited seminar at the MIT-Lincoln Laboratory, June 15, 1984 (also a meeting on cooperation under the AFSOR program).
7. "Solid State Epitaxial Growth of Single Crystal NbN on Sapphire," J. R. Gavalier, J. Gregg, and J. Schreurs, contributed paper at 17th Int. Conf. on L. Temp. Phys., Karlsruhe FRG (August 1984).
8. "Reactively Sputtered  $V_3Si$  and  $Nb_3Ge$  Films," J. R. Gavalier and J. Gregg, contributed paper, Applied Superconductivity Conference, San Diego (September 1984).

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\*Speakers' names are underlined.

9. "Tunnelling and Interface Structure of Oxidized Metal Barriers,  
J. Talvacchio, A. I. Braginski, M. A. Janocko, and S. J. Bending,  
contributed paper, Applied Superconductivity Conference, San Diego  
(September 1984).



## 8. PATENTS AND INVENTIONS

1. "Method and Apparatus Utilizing Crystalline Compound Superconducting Elements Having Extended Strain Operating Range Capabilities Without Critical Current Degradation," J. W. Ekin, J. R. Gavaler, and A. I. Braginski, U. S. Patent.
2. "A Method for Fabricating High- $T_c$  Superconducting Tunnel Junctions," A. I. Braginski.

**END**

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